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DEVELOPMENT OF 1.06 MICRON HIGH EFFICIENCY
ANTIREFLECTIVE COATING

by

Fan Ruiying, Lu Yuemei



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By: Fan Ruiying, Lu Yuemei

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DEVELOPMENT OF 1.06 MICRON HIGH EFFICIENCY ANTIREFLECTIVE COATING
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Fan Ruiying Lu Yuemei

ABSTRACT

This article carries out analyses of errors associated with two layer antireflective coatings. It points out that two layer antireflective coatings associated with non $\lambda/4$ stacks have more stable optical properties than $\lambda/4$ stacks. Using Ta_2O_5 and SiO_2 to act as film materials, two layer antireflective coatings were prepared on K_9 glass substrate. The single surface reflectivity was below 0.03%, and laser damage threshold values were greater than 7 GW/cm^2 (laser light wave length 1.06 micron, pulse width 1 ns).

I. INTRODUCTION

In laser systems, aperture expansion telescopes in two stage laser amplifiers as well as various types of lenses all need to be plated with antireflective coatings. Low residual reflection, on the one hand, causes laser output optical power to very greatly increase. On the other hand, it is possible to prevent lens surface reflected light from giving rise to optical components being destroyed.

Single layer film antireflective results are limited by film materials themselves. It is not possible to reach very high, far from being able to satisfy high power laser system requirements. It is necessary to opt for the use of double layer or multilayer antireflective coatings. Due to the fact that it is only required to increase the transmission of a single wave length and considering simple and feasible technologies, selection was made of a double layer transmission increasing film system. In order to obtain relatively good technical

* Numbers in margins indicate foreign pagination.
Commas in numbers indicate decimals.

replicability, we carried out error analyses of standard $\lambda/4$ double layer films and non $\lambda/4$ double layer films. Selection was made of non $\lambda/4$ double layer films associated with relatively large optical thickness tolerances. In conjunction with this, experimental research was carried out on film materials. Technical conditions were found capable of achieving high efficiency transmission increases and guaranteeing preparation replicability.

II. FILM SYSTEM SELECTION AND ERROR ANALYSIS

When light is vertically incident on transparent substrate plated with two layer coatings (for example, Fig.1), the reflection rate R can be determined from the equation below:

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$$R = \frac{\left\{ r_1^2 + r_2^2 + r_3^2 + r_1^2 r_2^2 r_3^2 + 2r_1 r_2 (1+r_3^2) \cos 2\phi + 2r_2 r_3 (1+r_1^2) \cos 2\psi \right.}{\left. + 2r_1 r_3 \cos 2(\phi + \psi) + 2r_1 r_2^2 r_3 (\phi - \psi) \right\}}{\left\{ 1 + r_1^2 r_2^2 + r_1^2 r_3^2 + r_2^2 r_3^2 + 2r_1 r_2 (1+r_3^2) \cos 2\phi + 2r_2 r_3 (1+r_1^2) \cos 2\psi \right.}{\left. + 2r_1 r_3 \cos 2(\phi + \psi) + 2r_1 r_2^2 r_3 \cos 2(\phi - \psi) \right\}}$$

In the equation, r_1 , r_2 , and r_3 are Fresnel coefficients:

$$r_1 = \frac{n_0 - n_1}{n_0 + n_1}, \quad r_2 = \frac{n_1 - n_2}{n_1 + n_2},$$

$$r_3 = \frac{n_2 - n_3}{n_2 + n_3},$$

ϕ and ψ are phase differences associated with various layers,

$$\phi = \frac{2\pi n_1 d_1}{\lambda}, \quad \psi = \frac{2\pi n_2 d_2}{\lambda}.$$

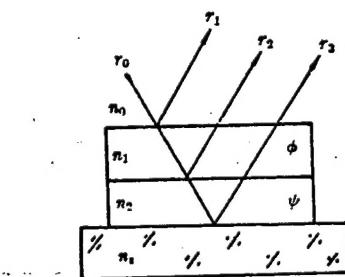


Fig.1 Two Layer Film Model

in situations where refraction indices are accurately determined, so long as one adjusts the thickness of the two layers, that is, varies ϕ and ψ , it is possible to make reflection rates associated with film systems be zero. During ordinary plating processes, one always makes the inside coating layer adjacent to the substrate first use high refractive index film material n_2 to coat to a certain reflectivity value R_m , making the phase thickness of the layer in question satisfy ψ . Next, low refractive index film material n_1 is plated to an extreme value. Following that, it again returns to the second extreme value. At this time, the phase thickness of the low refractive index layer is ϕ . Because of this, this film layer which completes coating the first extreme value is, in actuality, composed of a synthesis of two types of film material. The refractive indices of the film layers in question can be equivalent to the refractive index n_e associated with a single film layer (for example, as shown in Fig.2). For convenience, we name it the equivalent layer.

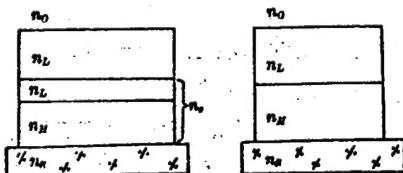


Fig.2 $\lambda/4$ and Non $\lambda/4$ Two Layer Film

From calculation formulae for two layer films, it is possible to obtain that fact that, when $n_0=1$, $n_s=1.506$, $n_L=1.45$, and $n_H=2.04$, so long as $R_m=10.7\%$, it is then possible to obtain zero reflection at center wave length locations. At this time, $n_e=1.782$. Using $\lambda/4$ two layer films associated with $n_H=n_e=1.782$, $n_L=1.45$, it is also possible to obtain the same type of structure. Taking these two types of film systems, error analysis comparisons are carried out as follows.

First of all, consider the situation when--in these two types of film systems--film material indices of refraction

deviate from the matching values which correspond to obtaining zero reflection. When they possess the same relative errors, their influences on overall reflectivity spectra are as shown in Tables 1 and 2.

TABLE 1 WHEN n_H DEVIATES FROM OPTIMUM VALUES + OR - 7.85% AND $n_L=1.45$ DOES NOT VARY

1 非 $\lambda/4$ 双层膜			2 $\lambda/4$ 双层膜		
n_H	R_{\min}	g_{\min}	n_H	R_{\min}	g_{\min}
2.04	0	1.0	1.782	0	1.0
2.20	0.0069%	1.0	1.922	0.57%	1.0
1.88	0.016%	1.0	1.642	0.66%	1.0

Key: (1) Non $\lambda/4$ Two Layer Film (2) $\lambda/4$ Two Layer Film

TABLE 2 WHEN n_L DEVIATES FROM OPTIMUM VALUES + OR - 6.9% AND $n_H=2.04$ AS WELL AS $n_e=1.782$ DO NOT VARY

1 非 $\lambda/4$ 双层膜			2 $\lambda/4$ 双层膜		
n_L	R_{\min}	g_{\min}	n_L	R_{\min}	g_{\min}
1.45	0	1.0	1.45	0	1.0
1.55	0.28%	1.01	1.55	0.44%	1.0
1.35	0.35%	0.985	1.35	0.51%	1.0

3 注: $g_{\min} (= \lambda_0 / \lambda_{\min})$ 为反射率极小值对应的相对波数。

Key: (1) Non $\lambda/4$ Two Layer Film (2) $\lambda/4$ Two Layer Film
 (3) Note: $g_{\min} (= \lambda_0 / \lambda_{\min})$ is the relative wave number corresponding to minimum reflectivity values.

From Tables 1 and 2, it is possible to see that, in non $\lambda/4$ two layer films, when film material refraction indices deviate from matching values, their influences on reflection rate minimum values, in all cases, are smaller than in two layer $\lambda/4$ films. In particular, high refractive index film material differences are more obvious.

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Next, analyzing the influences of the two types of film system reflectivity control errors (in situations where indices of refraction are invariable, it then reflects thickness errors) on overall reflection rate spectra characteristics, it was discovered that, comparing thickness error influences to refraction index error influences, they are not worth mentioning. Therefore, overall error influences are primarily given rise to by film material refractive indices (Table 3 takes non $\lambda/4$ two layer films as an example and analyzes situations when interior and exterior layers, respectively, possess the same relative control deviations). Because of this, it is possible to conclude that non $\lambda/4$ two layer film systems will be more stable than $\lambda/4$ two layer film systems. Moreover, non $\lambda/4$ film systems suffer less from restrictions associated with film material refractive indices. Therefore, we selected them to act as experimental film systems. The actual system was: $A(\alpha_1 L, \alpha_2 H)$ $G(\alpha_1 \text{ and } \alpha_2 \text{ are thicknesses relative to } \lambda/4, \alpha_2 < 1, \alpha_1 > 1)$. Calculations were made when $n_o=1.0$, $n_s=1.506$, $n_L=1.45$, and $n_H=2.04$. Phase thicknesses associated with the two layers were $\delta_L = 1.9967$ (radian) and $\delta_H = 0.6059$ (radian). Their spectral curves are seen in Fig.3. Center wave length is located at $R_{\min}=0$.

TABLE 3 INFLUENCES OF REFLECTIVITY CONTROL DEVIATIONS ASSOCIATED WITH NON $\lambda/4$ TWO LAYER FILMS

1 内层膜			2 外层膜		
$(\frac{\Delta R}{R})_H$	R_{\min}	g_{\min}	$(\frac{\Delta R}{R})_L$	R_{\min}	g_{\min}
$\frac{1}{220}$	0.00001%	1.0	$\frac{1}{220}$	0.005%	0.975
$\frac{1}{110}$	0.00007%	1.0	$\frac{1}{110}$	0.008%	0.965
$\frac{1}{55}$	0.00029%	1.0	$\frac{1}{55}$	0.019%	0.940

Key: (1) Inner Layer Film (2) Outer Layer Film

Looking at Tables 1-3 as a whole, it is possible to see that, in $\lambda/4$ two layer films, it goes without saying, there are influences that come with deviations in refractive indices or thicknesses. In all cases, it is more obvious in outer layer films than inner layer films. The explanation for this is nothing else than the importance of strictly controlled outer layer films in $\lambda/4$ two layer film preparation.

III. PREPARATION TECHNIQUES

1. Film Material Research

(a) Ta_2O_5 Film Material Characteristics

We opted for the use of compressed pieces of white Ta_2O_5 powder with 99.95% purity. Going through 900°C high temperature furnace sintering for 8 hours, pieces of Ta_2O_5 material were prepared. Using E model gun evaporation techniques, studies were done of Ta_2O_5 film material refractive indices following changes

in technical conditions as well as the status of its chromatic dispersion. Tables 4-7, respectively, clearly show refractive indices following changes in technical conditions as well as chromatic dispersion.

TABLE 4 n FOLLOWING CHANGES IN SUBSTRATE TEMPERATURE

$t(^{\circ}\text{C})$	250	280	330
n	2.067	2.077	2.083

In Table 4, data is all obtained under conditions with an oxygen charging pressure of 2×10^{-4} Torr. and deposition speeds of 7-8 Å/sec.

TABLE 5 n FOLLOWING CHANGES IN OXYGEN PRESSURE

$P(\text{托})$ (1)	2×10^{-4}	3×10^{-4}	4×10^{-4}
n	2.071	2.058	2.048

Key: (1) Torr.

Table 5 data was all obtained with substrate temperature 280°C , deposition speed 7-8 Å/sec, and an initial gas pressure of 1×10^{-4} Torr.

TABLE 6 n FOLLOWING CHANGES IN DEPOSITION SPEED

$v(\text{Å/秒})$ (1)	4~5	9
n	2.060	2.065

Key: (1) Second

Table 6 data was all obtained with substrate temperature 280°C and gas pressure at 3×10^{-4} Torr.

The data discussed above is all average values associated with multiple iterations of experiments. n values are given with $\lambda = 6328 \text{ \AA}$. Measurements were made on TP-77 model elliptical deviation thickness measuring devices.

TABLE 7 CHROMATIC DISPERSION ASSOCIATED WITH Ta_2O_5 REFRACTIVE INDICES

λ (微米)	1.05	0.94	0.82	0.75	0.65	0.54	0.46
n	2.05	2.053	2.056	2.061	2.066	2.09	2.11

Key: (1) Microns

Table 7 data is the result of going through multiple interactions of experiments with the same substrate temperature (300°C), oxygen pressure (1.6×10^{-4} Torr.), and deposition speed (8 \AA/sec).

From the experiments described above, it is possible to see that Ta_2O_5 material refractive indices do not vary greatly within ranges at fixed temperatures, gas pressures, and deposition speeds.

Results from measurements of scattering rates in the vicinity of 6500 \AA for single layer Ta_2O_5 thin films on K_9 substrate clearly show that its scattering losses are equivalent to ZrO_2 . However, absorption losses are very small.

Experimentation discovered that, under the same type of deposition conditions, absorption coefficients associated with thin films obtained from freshly evaporated Ta_2O_5 material and

Ta_2O_5 material which had been used for evaporation multiple times were different. The latter clearly increased. The key thing is Ta_2O_5 material decomposition during evaporative coating processes. The ratio of released oxygen and Ta molecules varies. This leads to Ta_2O_5 components remaining in crucibles changing from those produced. Because of this, each iteration of evaporative coating adds some new material or enlarges the amount of oxygen charging providing compensation.

(b) SiO_2 Film Material Characteristics

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Large amounts of experimentation have already demonstrated that SiO_2 film material has relatively good reproducibility of refractive indices in air. However, is the situation in a vacuum the same or not? To this end, a series of experiments were also carried out on SiO_2 film material, respectively, making it under different technical conditions associated with $\lambda/4$ single layer film ($\lambda = 6328 \text{ \AA}$). Changes in it were observed before and after heat treatment in a vacuum and in air (drying temperature was always 280°C). Concrete data is set out in Tables 8-10.

TABLE 8 RELATIONSHIPS OF n TO DEGREES OF VACUUM

气压 P (托)	3×10^{-4}	2×10^{-4}	$6 \sim 4 \times 10^{-5}$
n	1.359	1.372	1.40

Key: (1) Gas Pressure P (Torr.)

Table 8 data was obtained on substrate without the addition of drying. Deposition speeds were basically maintained at 45 \AA/sec.

TABLE 9 RELATIONSHIPS OF n TO SUBSTRATE TEMPERATURE T

		T (°C)		
		室温 30°C	225°C	275°C
		n		
3	真空 2×10^{-4} 托	1.372	1.389	1.403
4	5 烘烤前	1.453	1.455	1.462
	6 烘烤后	1.447	1.448	1.449

Key: (1) Ambient Atmosphere (2) Room Temperature (3) Vacuum 2×10^{-4} Torr. (4) In Atmosphere (5) Before Drying (6) After Drying

During Table 9 sample preparation, degree of vacuum was maintained at 2×10^{-4} Torr. and deposition speed was approximately 45 Å/sec.

TABLE 10 RELATIONSHIPS OF n TO DEPOSITION SPEED v

		v (Å/秒)		
		18	45	72
		n		
3	真空 (2×10^{-4} 托)	1.355	1.372	1.392
4	5 烘烤前	1.445	1.453	1.457
	6 烘烤后	1.441	1.447	1.447

Key: (1) Ambient Atmosphere (2) v (A/Sec) (3) Vacuum (2×10^{-4} Torr.) (4) In Atmosphere (5) Before Drying (6) After Drying

During Table 10 sample preparation, substrate was not heated, and degree of vacuum was maintained at 2×10^{-4} Torr.

From Tables 8-10, it is possible to see that: (1) SiO_2 refractive indices follow rises in substrate temperature, degree of vacuum and deposition speed and increase; (2) SiO_2 refractive indices follow changes in deposition conditions and are more sensitive in a vacuum than in the atmosphere; (3) SiO_2 refractive index changes from vacuum \rightarrow atmosphere \rightarrow after drying is a reversible process, that is, in a vacuum, they are relatively low. Put into the atmosphere, they clearly go up. And, after going through drying, they again slightly drop.

2. Key Preparation Points

Preparation of the antireflective coatings in question did not adopt some special techniques. In order to obtain good preparation reproducibility, it was necessary to pay attention to the technical conditions when film material is deposited--for example, substrate temperature, oxygen pressure, deposition speed, and so on (in particular, SiO_2 film material refractive index changes are relatively sensitive in a vacuum, moreover, its influence on antireflective coatings is also relatively great). Only then is it possible to obtain refractive indices with relatively stable numerical values. At the same time, it is necessary to pay attention to the differences between film material refractive indices in a vacuum and in the atmosphere, seeking out the δ'_H value in a vacuum (it is slightly smaller than designed δ_H values). Only then is it possible to make prepared antireflective coatings achieve spectral properties relatively in consonance with design values.

IV. EXPERIMENTAL RESULTS

1. Spectral Properties

As far as plated samples are concerned, the results from making reflection rate point measurements on low reflectivity measurement devices as well as from making scanning measurements on UV-360 spectral instruments are set out in Table 11 and Fig.3. Results clearly show that the antireflective coatings in question, in the vicinity of 1.06 micron wave lengths, have single surface reflectivities associated with K_g substrates of $R \leq 0.03\%$.

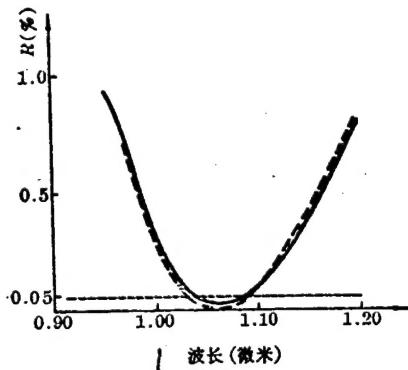


Fig.3 Comparison of Experimental Results and Theoretical Calculations

— Actual Measurement Curve - - - Theoretical Curve
Key: (1) Wave Length (microns)

Use was made of scattering measurement equipment to determine the scattering rate associated with antireflective film of $\lambda = 0.65$ micron to be $1 \sim 1.5 \times 10^{-4}$. A good deal lower than the scattering associated with single layer Ta_2O_5 film.

2. Laser Damage Threshold Values

Using 1.06 micron pulse width for 1 ns pulse lasers, it was determined that the damage threshold value for the antireflective coatings in question is 7-8 GW/cm². This is able to satisfy the requirements of high power laser systems.

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TABLE 11 LOW REFLECTIVITY MEASUREMENT DATA ASSOCIATED WITH ANTIREFLECTIVE COATINGS

1) 样 品 号	2) λ (微米)		
	1.065	1.061	1.055
	R(%)		
1	0.017	0.016	0.015
2	0.021	0.016	0.010
3	0.024	0.019	0.018
4	0.013	0.015	0.018
5	0.013	0.014	0.013
6	0.020	0.012	0.010
7	0.010	0.010	0.011
8	0.016	0.011	0.010
9	0.010	0.011	0.012
10	0.026	0.018	0.020

Key: (1) Sample No. (2) Micron

In the area of low reflectivity spectral measurements, Professors Maziwaru Ooki and Kiroiki Tatashi gave vigorous support. For this, we take the opportunity to express our thanks.

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